October 20, 1982

Michael J. Hazel
Regulatory Compliance Section
Solid and Hazardous Waste Division
Minnesota Pollution Control Agency
1935 West County Road B2
Roseville, Minnesota 55113-2785

Dear Mike:

The HPLC analytical results for the PNA samples received on September 21, 1982, are summarized in Table 1. The 4-L samples did present a problem in that the standard sample size for Method 610 is 1 liter, not 4 liters. Each 4-L sample was placed in a 6-L separatory funnel and extracted with three 200-mL aliquots of methylene chloride. A 1-liter spiked distilled water sample was included with the sample set to verify that the change in sample size did not adversely affect the results. The dosing solution was prepared from NBS Standard Reference Material 1647. Both the 4-L well water and the 1-L distilled water samples were spiked with identical amounts of the PNAs. The results given in Table 2 indicate that in either case, 4 liters extracted with 3 x 200 mL methylene chloride or 1 liter extracted with 3 x 60 mL methylene chloride; the recoveries parallel each other. However, in the future please send 1-liter samples.

The GC/MS samples were dosed at a level 10 times that used for the HPLC samples. In the HPLC samples, the dosing level was not high enough for the first 8 eluting PNAs -- in all but one case, the background level was larger than the dose level. The dosing level was high enough for the last eight eluting PNAs -- in all cases the background level was less than the dose level. The recoveries for the last eight eluting PNAs averaged 148% and 136% for the well water and distilled water respectively.

Future analyses will utilize redistilled methylene chloride. This should reduce the levels found in the Field Blank. The Method Detection Limit for this HPLC analysis also should be estabished using the dosing levels and analytical conditions for this set of samples.

After concentration, the sorbent is changed to acetonitrile and further concentrated to 0.5 mL. Water is added to give a 1.0 mL final extract of a 50:50 acetonitrile/water solution. Analysis is performed using a 100 µL injection of this extract. Standards injected are also in a 50% acetonitrile/water solution.

The GC/MS results will be sent when available.

Sincerely yours,

Denis Foerst, Reseach Chemist
Organic Analyses Section
Physical and Chemical Methods Branch

Enclosure:
Tables 1 and 2

cc: Mike Kosakowski (w/enclosures)

Table 1

PNA Concentrations in ng/L[a]

Volumes of Sample (L)	Detector	SLP-4 82-01 3.81	SLP-4 82-02 3.82	SLP-7 82-04 3.86	SLP-15 82-05 3.90	Field Blank 82-06 4.00
Naphthalene	UV	b	b	b	b	b
Acenaphthylene	UV	b	b	b	b	b
Acenaphthene	F	63.2	80.2	152	8670	ND
Fluorene	F	2.6	3.9	10.2	350	ND
Phenanthrene	F	24.0	29.8	3.0	107	1.4
Anthracene	F	3.0	4.4	4.7	233	ND
Fluoranthene	F	11.0	5.3	1.8	374	0.94
Pyrene	F	7.5	3.5	8.9	339	0.36
Benzo(a)anthracene	F	0.81	0.34	0.24	19.0	0.10
Chrysene	F	0.60	0.0[c]	0.40	11.6	0.57
Benzo(b)fluoranthene	F	0.12	0.0[c]	0.0[c]	0.0[c]	0.49
Benzo(k)fluoranthene	F	0.0[c]	0.0[c]	0.0[c]	0.0[c]	0.66
Benzo(a)pyrene	F	0.48	0.42	0.18	0.07	ND
Dibenzo(a,h)anthracene	F	0.0[c]	0.0[c]	0.05	0.0[c]	0.09
Benzo(g,h,i)perylene	F	0.0[c]	0.0[c]	0.16	0.10	0.26
Indeno(1,2,3-cd)pyrene	F	0.07	0.07	ND	0.14	ND

[a]Results corrected by field blank.

[b]Not detected by qualitative criterion of ratio of signal at 230 and 254 nm.

[c]Amount in field blank greater than amount in sample.

ND - Not detected.

Table 2
Recovery of PNAs in Well Water and Distilled Water

Compound	Spike Level in ng	SLP-4[a] ng Found in Spiked Sample	SLP-4[a] ng Found in Background Sample[c]	SLP-4[a] Recovery (%)	Distilled Water[b] ng Found in Spiked Sample	Distilled Water[b] ng Found in Background Sample	Distilled Water[b] Recovery (%)
Naphthalene	225	d	d	d	d	d	d
Acenaphthylene	19.1	d	d	d	d	d	d
Acenaphthene	21.0	405	306	471	78.8	105	0
Fluorene	4.9	68.9	14.8	1100	24.6	0.0	502
Phenanthrene	5.1	138	119	373	31.6	25.3	124
Anthracene	3.3	18.0	16.7	39	14.0	38.4	--
Fluoranthene	10.1	28.2	24.1	41	7.5	18.7	--
Pyrene	9.8	27.3	14.7	129	12.7	9.8	30
Benzo(a)anthracene	5.0	6.6	1.7	98	5.7	0.4	106
Chrysene	4.7	13.0	2.3	228	9.4	3.1	134
Benzo(b)fluoranthene	5.1	9.5	1.8	151	8.2	1.2	137
Benzo(k)fluoranthene	5.0	10.1	1.8	166	8.8	1.4	148
Benzo(a)pyrene	5.3	4.6	1.6	57	4.0	0.3	70
Dibenzo(a,h)anthracene	3.7	5.4	0.4	135	4.6	0.0	124
Benzo(g,h,i)perylene	4.0	11.3	0.8	263	9.0	0.0	225
Indeno(1,2,3-cd)pyrene	4.1	3.7	0.3	83	6.5	0.7	142

[a] 4-liter sample extracted with 3 x 200 mL methylene chloride

[b] 1-liter sample extracted with 3 x 60 mL methylene chloride

[c] Sample 82-02

[d] Not detected, by qualitative criterion of ratio of signal at 230 and 254 nanometers.